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Graphite/PMR Polyimide Composites with Improved Toughness

(NASA-TM-87081) GRAPHITE/PMR POLYIMIDE
COMPOSITES WITH IMPROVED TOUGHNESS (NASA)
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GRAPHITE/PMR POLYIMIDE COMPOSITES WITH IMPROVED TOUGHNESS

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1. ABSTRACT

E-2661
Studies were conducted to determine the toughness of composites made with modified PMR polyimides and Celion 6000 graphite fibers. Various types/levels of monomer reactants containing flexible links were incorporated into PMR resin compositions used to prepare composites. The composites were evaluated for toughness using instrumented drop weight and 10° off-axis tensile tests at room temperature, and for strength using flexure and short-beam shear tests at room temperature and at elevated temperature. The effect of resin composition on composite processability, thermo-oxidative stability, toughness and mechanical properties are discussed.

2. INTRODUCTION

Addition curing PMR polyimides are now gaining wide acceptance as matrix resins for high temperature composite engine applications. Considerable cost and weight savings can be achieved by using fiber reinforced polyimide composites in place of metals currently used in

the moderately high temperature zones (up to 600 °F) of jet engines. The technology for application of these advanced composite materials to jet engines is developing rapidly. General Electric Co., under Navy and NASA sponsorship, has recently completed the development of a graphite/PMR polyimide air by-pass duct to replace the titanium duct currently used on the G.E. F-404 engine which powers the Navy's F-18 fighter. Other components currently under consideration for composite applications include: engine inlet guide vanes, first and second stage stators and anti-icing leading edge wing panels.

A major concern in all of the above applications is the material's resistance to low velocity impact damage. Low velocity impact can result in "hidden" internal damage in composite structures.⁽¹⁾ While, initially, the damage may not degrade the structural integrity of the component, it is possible that load reversals from vibrations induced in the structure under flight conditions can cause the

Keywords: PMR polyimide;
Composites; Toughness

damage to grow until structural failure occurs. This type of damage which can result from dropped tools, runway debris, etc., can occur in both the airframe structures and the engine itself. With the broadening interest to improve engine performance with composites, the need for tougher, more damage tolerant high temperature composite materials is obvious.

PMR polyimides, which exhibit excellent elevated temperature oxidative resistance and mechanical properties, are inherently brittle matrix resins. Improving the strain capability of PMR polyimide, without significantly affecting composite stiffness and glass transition temperature (T_g), should result in a more damage resistant or damage tolerant PMR composite material. This report describes a study in which flexible connecting links were introduced into the backbone of PMR resins used to fabricate graphite fiber PMR composites. The purpose of this investigation was to determine the effect of the flexibilized resin structure on the toughness, physical and elevated temperature mechanical properties, mechanical properties retention at elevated temperatures, and processability of graphite fiber reinforced PMR composites. The test methods used to assess the effect of resin molecular structure on composite properties included: composite

flexural and interlaminar shear, Charpy and low velocity drop weight impact, 10° off-axis intralaminar shear, and TMA, T_g measurements.

3. EXPERIMENTAL PROCEDURE

3.1 Preparation of PMR Solutions

The monomer reactants used to prepare the resins used in this study are shown in Table I. All of the monomers, except the dimethylesters, were purchased from commercial sources. The dimethylesters, of 3,3',4,4'-benzophenonetetracarboxylic acid (BTDE), and 1,2,4,5-benzenetetracarboxylic acid (PMDE), were prepared as 50 wt % solutions by refluxing a suspension of the corresponding dianhydride in anhydrous methanol until all the solids had dissolved and then for an additional 2 hr.

PMR reactant solutions were prepared at room temperature by dissolving the dimethylester and diamine monomers in a calculated amount of anhydrous methanol to yield a fully mixed monomer solution of 50 wt % solids, and then adding the NE monomer. With one exception, the monomer stoichiometry used for the PMR solutions was 2 NE/2.087 dimethylester/3.087 diamine.

3.2 Composite Fabrication

All composites were prepared from prepreg tape made with unsized Celion 6000 graphite tow material. The prepreg tape was prepared by brush application of the resin

solution onto drum wound (12 turns/in) unidirectional tows to yield composites having a nominal 58 vol % fiber after curing. The prepreg was then dried on the rotating drum under a quartz heat lamp to a volatile content of 12-13 wt %. The tapes were then removed from the drum and then cut and stacked according to predetermined orientations and thicknesses required for fabrication of composites for each of the tests to be performed. The prepreg stacks were then staged under 0.1 psi pressure in preforming tools for 60 min at 400 °F. Composites were then compression molded by placing the staged prepreg layup into a flat matched-metal die at room temperature, inserting the die into a platen press preheated to 600 °F, and then applying contact pressure. When the die reached 450 °F, 500 psi pressure was applied. Composites exhibiting reduced resin flow under 500 psi cure pressure (those containing either PMDE or the diamine mixture of BDAF/PPDA at a 3:2 molar ratio) were molded under 1000 psi pressure. When the die reached 600 °F, pressure and temperature were maintained for 90 min. The press heaters were then turned off and the composites allowed to cool slowly (~1 hr) to 450 °F under pressure. The pressure was then released and the composites were cooled at 20 °F/min to room temperature. The composites were then

given a free standing postcure in air at 600 °F for 24 hr.

4. COMPOSITE TESTING

Prior to testing, all composites were inspected for porosity using an ultrasonic C-scan technique.

4.1 Flexural and Interlaminar Shear Strength Tests

Flexural and interlaminar shear strength tests were performed on composites having a nominal thickness of 0.090 in. Flexural strength tests were performed on specimens 0.25 in wide by 2.5 in long in accordance with ASTM D-790 at a constant span/depth of 28. Interlaminar shear strength tests were performed on 0.25 in wide specimens, essentially in accordance with ASTM D-2344 at a span/depth of five. Elevated temperature tests were performed in an environmental heating chamber. Forced convection air ovens were used for long-term isothermal exposure at 600 °F. The air change rate was 0.061 in³/min. Composite weight measurements were made throughout the exposure time period. Property values reported are averages of three to six tests.

4.2 Charpy Impact Tests

Unnotched Charpy impact tests were performed on specimens cut from 28 ply unidirectionally reinforced composites having a nominal thickness of 0.220 in. The specimen dimensions were 0.20 in wide by 2.5 inches long with the direction

of the reinforcement parallel to the long dimension. Values reported are averages of five or more tests.

4.3 Ten Degree Off-Axis Tensile Tests

Ten degree off-axis tensile tests were performed on five ply composites having a nominal thickness of 0.040 in. Specimens were 0.5 in wide by 8 in long with the reinforcement oriented 10° off the 8 in, or load direction. Strain measurements were determined in the zero or load direction, using a clip-on extensometer. The load rate was 0.05 in/min. Shear properties were calculated from the 10° off-axis tensile data as described in Ref. 2. Values reported are averages of five tests.

4.4 Instrumented Drop Weight Impact Tests

Drop weight tests were performed on 4 in x 4 in x 0.090 in 12-ply composites having a balanced 0°/90° layup. The tests were performed on a Dynatup instrumented drop weight tester. Two tests were performed on each material. The test specimens were clamped on all four sides.

5. RESULTS AND DISCUSSION

The structures of the monomer reactants used in this study are shown in Table I. PMR-15 resins are prepared from NE, MDA and BTDE. The idealized structure of the end-capped polyimide oligomer of PMR-15 resin is shown in Fig. 1. Also shown in Fig. 1 are the repeat unit

structures of NE/BTDE/ BDAF, and NE/PMDE/BDAF. The value of n for PMR-15 and NE/BTDE/BDAF is 2.087. The n value for NE/PMDE/BDAF is 2.0. It can be seen that by substituting BDAF for MDA, the number of flexible connecting groups in the repeating unit is increased from two to three. By substituting PMDE and BDAF for BTDE and MDA, respectively, the number of flexible connecting groups remains the same. The effect of these changes in the resin molecular structure on composite impact energy is shown in Table II. The table compares the unnotched Charpy impact energies to failure of unpostcured unidirectionally reinforced composites made with unsized Celion 6000 graphite fiber. Also shown are the composite fiber volumes and unpostcured T_g 's. Unpostcured specimens were used in these initial screening tests so that the results would not be influenced by oxidative cross-linking which can occur during postcure in air.

The results of the Charpy impact tests show that the substitution of BDAF for MDA results in a 35 percent increase in impact energy over that of the baseline PMR-15 resin composite. However, the toughened composite exhibited a glass transition temperature (T_g) of 509 °F compared to 555 °F for that of the PMR-15 composite. The composite prepared with the resin containing the flexible BDAF

diamine and PMDE exhibited the highest T_g (572 °F) and a 13.4 percent improvement in impact energy over the baseline material. The higher T_g exhibited by the PMDE/BDAF composite results from the planar, stiffer structure of PMDE. The concomitant increase in T_g and impact energy shown for the PMDE/BDAF composite was unexpected. However, due to the lower resin flow exhibited by this material during curing, the PMDE/BDAF composites were cured under 1000 psi pressure while the PMR-15 and BTDE/BDAF composites were cured under 500 psi of pressure. The higher cure pressure and 2 percent lower fiber volume (56 versus 58 vol % for the PMR-15 and BTDE/BDAF composites) may have contributed to the higher than expected impact strength shown for the PMDE/BDAF composite.

A comparison of the room temperature and 600 °F mechanical properties of these composite materials, after a free standing 24 hr postcure at 600 °F, is shown in Table III. Also shown are the composite T_g 's after postcure and composite weight loss and retention of 600 °F mechanical properties after 1500 hr, 600 °F air exposure, for the PMR-15 and PMDE/BDAF composites. It can be seen that the resin modifications had no appreciable effect on room temperature mechanical properties. However, the resin modifications adversely

affected the elevated temperature composite properties due to the significantly lower T_g 's exhibited by composites containing BDAF. Because of the low T_g exhibited by the postcured BTDE/BDAF composite, only short-time 500 °F mechanicals are shown for this material. The differences in initial 600 °F mechanical properties between the PMR-15 and PMDE/BDAF composites can be attributed to the lower T_g (36 °F lower) shown for the PMDE/BDAF composite. However, while the PMR-15 composite retained greater than 90 percent of initial flexural and interlaminar shear strength properties, these same properties were more than 30 percent lower for the PMDE/BDAF composite which exhibited thermoplastic characteristics during 600 °F testing. In addition, the weight loss after 1500 hr exposure was 30 percent higher for the PMDE/BDAF composite over that of the baseline PMR-15 material.

The differences in the elevated temperature properties between the BDAF and PMR-15 composites can be attributed to different thermooxidative reactive mechanisms experienced by the two materials. The increase in T_g after postcure for the composites containing BDAF were significantly lower than that shown for the PMR-15 composite. While the PMR-15 composite T_g increased by 107 °F, the composites made with BTDE/BDAF and PMDE/BDAF

shear strain to failure (25 percent and 12.5 percent for the composites containing 4:1 and 3:2 mole ratios of BDAF/PPDA, respectively) over that shown for baseline PMR-15 composite. In contrast, the composites containing BTDE/MDA and all BDAF exhibited increases in strain to failure of 75 and 88 percent, respectively, over that shown for the baseline material.

Table IV lists the mechanical properties of the Celion 6000 reinforced BDAF/PMR composites discussed in Fig. 2. Listed are the initial flexural strengths and moduli and interlaminar shear strengths of the composites at room temperature and at 600 °F. The retention of 600 °F properties as well as weight loss after 1500 hr exposure to air at 600 °F is also shown. The results of the room temperature tests indicate that the use of the more flexible BDAF diamine in PMR composites has little or no effect on room temperature flexural and interlaminar shear strength properties. The lower initial 600 °F mechanical properties of the BDAF composites and the PMR-15 composite reflect the lower T_g 's exhibited by the BDAF composites. However, after 1500 hr exposure at 600 °F the flexural strength of the composite made with MDA/BDAF increased by 36 percent to a level only 5 percent lower than that shown for the baseline PMR-15 composite

while retaining 90 and 93.5 percent of its initial 600 °F flexural modulus and interlaminar shear strength, respectively.

It is interesting to note that while the MDA/BDAF composite exhibited an increase in flexural strength after 1500 hr exposure to air at 600 °F, the composites containing the BDAF/PPDA diamines did not exhibit any increase in 600 °F mechanical properties after the same exposure. These results support the hypothesis stated earlier regarding the beneficial effect of elevated temperature interactions between the methylene connecting group in MDA and the cross-linked structure from the nadic end-cap having a beneficial effect on long term elevated temperature properties retention of PMR composite materials.

Based on the 1500 hr, 600 °F weight losses exhibited by the PMR-15 control composite and the composites containing the mixed diamines, it appears composite weight loss is proportional to the molar ratio of BDAF present in the matrix resin of the composite. The composites containing 20, 60, and 80 mole % of the BDAF diamine exhibited 13.7, 16.5 and 20 percent weight loss, respectively, after 1500 hr exposure at 600 °F, compared to 10 percent weight loss for that of the baseline PMR-15 composite after the same exposure. This suggests that in order to retain oxidative

stability, low levels of the less oxidatively stable, flexible, BDAF monomer should be used to improve the toughness of PMR matrix resins.

The results of the drop weight impact tests on balanced 0/90 cross-ply composites are shown in Table V. Shown are the maximum load, the total energy absorbed during impact and the deflection at maximum load for each impacted specimen. The data shows that all of the BDAF toughened composites sustained a considerably higher impact load than the PMR-15 composite. The total energy absorbed values are also significantly higher for the toughened composites with the exception of the composite made with BDAF/PPDA at a 3:2 molar ratio. The composite made with resin containing MDA/BDAF at a 4:1 molar ratio sustained both the highest impact load and total energy of all the composites tested. In comparing the values for composite deflection at maximum load, it can be seen that the composite containing MDA/BDAF at a 4:1 molar ratio exhibited a 65 percent increase in deflection over that of the PMR-15 composite and a value only 7 percent lower than that shown for the all BDAF toughened composite.

Based on the results of the study shown so far, it can be seen that the matrix resin containing MDA/BDAF at a 4:1 molar ratio provides

composites with the best overall balance of toughness and elevated temperature properties retention.

Table VI provides a comparison of resin formulated molecular weight (FMW), composite T_g and composite ductility as determined by shear failure strain and deflection using the 10° off-axis tensile and drop weight impact tests, respectively. It is apparent that, for the PMR polymers investigated, improvements in composite ductility are obtained with some sacrifice in composite T_g . An effect of the reduced cross-link density of the higher FMW resins on composite T_g and ductility is not readily apparent. Although the higher FMW BDAF/PPDA resins contained a higher level of BDAF than the MDA/BDAF system, the expected improvements in composite toughness were off-set by the stiffening provided by the use of PPDA.

Finally, in comparing the results shown for composite 10° off-axis strain and maximum drop weight deflection shown in Table VI, there appears to be fairly close agreement between strain-to-failure and maximum deflection for the two tests. The results of these two tests are plotted in Fig. 3. This plot was constructed to determine if there might be a correlation between the two tests. Although there is some deviation between the lower and higher strained materials, the correlation appears to

increased by only 44 and 58 °F, respectively. In a recent study conducted at the Lewis Research Center,⁽²⁾ it was found that PMR-15 resin composites containing MDA exhibited less weight loss and higher retention of mechanical properties after extended exposure to air at 600 °F than PMR composites containing more thermally stable diamines. The superior elevated temperature performance of the MDA containing PMR-15 material was attributed to oxidative cross-linking between the methylene of MDA, present only in PMR-15, with the molecular structure formed during the addition reaction of the NE end-cap.

Thus, the lower initial 600 °F mechanical properties of the PMDE/BDAF composite compared to those shown for the PMR-15 composite can be attributed to the 36 °F lower T_g of the PMDE/BDAF composite, whereas, the lower 600 °F mechanical properties and 30 percent higher weight loss after 1500 hr exposure to air at 600 °F is probably due to the absence of favorable sites for cross-linking in the PMDE/BDAF composite.

The Charpy and T_g results indicate that the introduction of flexible connecting groups into the backbone of the PMR matrix has the desirable effect of improving composite toughness but the undesirable effect of reducing the upper use temperature of these materials.

To identify the resin composition which provided the best overall balance of composite toughness and T_g , composites were prepared with resins containing mixtures of BDAF and MDA or PPDA. This series of composites were screened for intralaminar shear properties using a 10° off-axis tensile test. Since the Charpy impact test is primarily a bending test and is heavily fiber dominated, it was felt that the 10° off-axis test, because it subjects the resin to pure strain,⁽²⁾ would provide a better indication of composite matrix strain capability.

Figure 2 shows the 10° off-axis shear properties of composites prepared from NE, BTDE and mixtures of the diamines BDAF/MDA and BDAF/PPDA, along with the baseline PMR-15 and all BDAF composites. These resins were formulated with a constant stoichiometry of 2.087, the same as PMR-15 resin, so that the number of imide groups would not influence the test results. The figure shows that all of the composite prepared with mixed diamines exhibited T_g 's lower than the PMR-15 composite. The figure also shows that replacing 20 mole % of the MDA with BDAF had the most pronounced combined effect of raising composite T_g and strain-to-failure. Composites containing higher ratios of BDAF along with the more planar PPDA exhibited slightly higher T_g 's but only marginal improvement in composite

be close enough to allow the use of the more simple 10° off-axis shear test for screening relative composite toughness.

6. CONCLUSIONS

Based on the results of this study, the following conclusions can be drawn:

- (1) The toughness of PMR-15 composites can be improved by incorporating aromatic diamines containing flexible connecting groups into the matrix resin.
- (2) Improvements in the toughness of PMR composites are accompanied with a reduction in use temperature.
- (3) Polymer matrix composites can be screened for toughness by the use of the relatively simple 10° off-axis tensile test.

7. REFERENCES

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2. C.C. Chamis and J.H. Sinclair: "10° Off-Axis Tensile Test for Intralaminar Shear Characterization of Fiber Composites," NASA TN-D-8215, (1976).
3. W.B. Alston: "Replacement of MDA with More Oxidatively Stable Diamines in PMR Polyimide," NASA CP-2385, (1983).

8.0 BIOGRAPHIES

8.1 Raymond D. Vannucci

Raymond D. Vannucci has been employed at NASA Lewis Research Center since 1965. He received a B.S. degree in Engineering from Cleveland State University. Presently he is working as a Materials Engineer in the Polymer Composites Branch. His current research involves the fabrication and characterization of polymer composites.

8.2 Kenneth J. Bowles

Kenneth J. Bowles received his Ph.D. in Materials Science from Case Western Reserve University. Since the start of his employment at Lewis Research Center in 1958, he has conducted research in the areas of liquid metal corrosion, advanced nuclear fuel element materials and composite materials. He is currently working as a Materials Engineer in the Composites Branch in the area of composite toughness.

TABLE I - MONOMER STRUCTURES

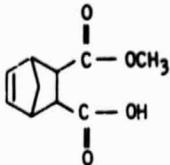
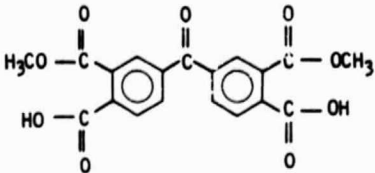
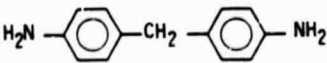
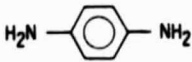
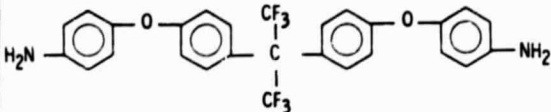
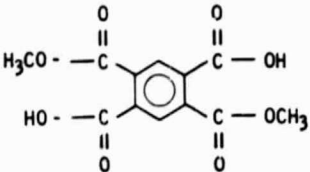
STRUCTURE	NAME	ABBREVIATION
	MONOMETHYL ESTER OF 5-NORBORNENE-2,3- DICARBOXYLIC ACID	NE
	DIMETHYL ESTER OF 3,3',4,4'- BENZOPHENONETETRACARBOXYLIC ACID	BTDE
	4,4'-METHYLENEDIANILINE	MDA
	P-PHENYLENEDIAMINE	PPDA
	BIS(AMINOPHENOXY) PHENYLHEXAFLUOROPROPANE	BDAF
	DIMETHYLESTER OF 1,2,4,5- BENZENETETRACARBOXYLIC ACID	PMDE

TABLE II - CELION 6000/BDAF-PMR COMPOSITE CHARPY IMPACT PROPERTIES^A

MATRIX RESIN	FIBER VOL. %	T _G ^B	IMPACT ENERGY, in-lb
NE/BTDE/MDA (PMR - 15)	58	550° F	37.1
NE/BTDE/BDAF	58	509° F	49.9
NE/PMDE/BDAF	56	572° F	42.1

^A UNNOTCHED
^B NO POSTCURE

TABLE III - CELION 6000/BDAF-PMR COMPOSITE MECHANICAL PROPERTIES

MATRIX RESINS	FIBER, vol. %	T _G ^(a) OF		RT	600° F	1200 hr AT 600° F
1. NE/BTDE/MDA (PMR-15)	58.0	662	FLEX. STR., ksi	244	140	135
			FLEX MOD., msi	16.5	15.2	12.5
			ILSS, ksi	15.0	7.2	7.0
			% wt. LOSS			10.0
2. NE/BTDE/BDAF	56.0	555	FLEX STR., ksi	240	123 ^(b)	
			FLEX MOD., msi	17.3	9.5	
			ILSS, ksi	14.5	6.7	
			% wt. LOSS			
3. NE/PMDE/BDAF	58.0	626	FLEX. STR., ksi	220	110	81
			FLEX. MOD., msi	17.5	14.9	8.0
			ILSS, ksi	13.8	6.2	4.5 (TP)
			% wt. LOSS			13.0

^(a) POSTCURED^(b) 500° F DATA

TP-Thermoplastic failure

TABLE IV - MECHANICAL PROPERTIES OF CELION 6000/BDAF-PMR COMPOSITES

RESIN ^(a) DIAMINE	T _G , °F	FLEX. STR., ksi/ MOD., msi		INTERLAMINAR SHEAR STR., ksi		AFTER 1500 hr AT 600° F		
		RT	600	RT	600	600° F FLEX. STR., ksi/MOD., msi	600° F INT. SHEAR STR., ksi	COMPOSITE WT. LOSS, %
PMR-15 CONTROL BTDE/MDA	662	24°/16.3	140/15.2	15.0	7.2	135/13.5	7.0	10.0
BTDE/MDA-BDAF (4:1)	612	255/17.8	94/11.2	15.7	6.1	128/10.0	5.7	13.7
BTDE/BDAF-PPDA (4:1)	615	239/16.0	86/11.0	15.9	4.9	85/8.5	4.3	20.0
BTDE/BDAF-PPDA (3:2)	630	242/17.0	100/12.5	14.2	6.6	87/10.0	5.1	16.5

(a) $n = 2.087$ FOR ALL RESINSTABLE V - DROP-WEIGHT IMPACT PROPERTIES OF
0/90 CELION 6000/BDAF-PMR COMPOSITES

DIAMINE	MAXIMUM LOAD, lb	TOTAL ENERGY, ft-lb	DEFLECTION AT MAX. LOAD, in
MDA (PMR-15)	640	9.80	.085
BDAF	742	11.44	.15
MDA/BDAF (4:1)	807	11.81	.14
BDAF/PPDA (4:1)	781	10.90	.12
BDAF/PPDA (3:2)	780	9.70	.10

TABLE VI - COMPARISON OF GLASS TRANSITION
TEMPERATURE AND COMPOSITE DUCTILITY

MONOMERS	FMW	T _G (°F)	10° OFF-AXIS STRAIN percent	MAX. DEFLECTION DROP WEIGHT, in
NE/BTDE/MDA (PMR-15)	1500	662	2.4	.085
NE/BTDE/BDAF-PPDA (3:2)	1976	630	2.7	.100
NE/BTDF/BDAF-PPDA (4:1)	2171	615	3.0	.120
NE/BTDE/MDA-BDAF (4:1)	1675	612	4.2	.140
NE/BTDE/BDAF	2418	555	4.5	.150

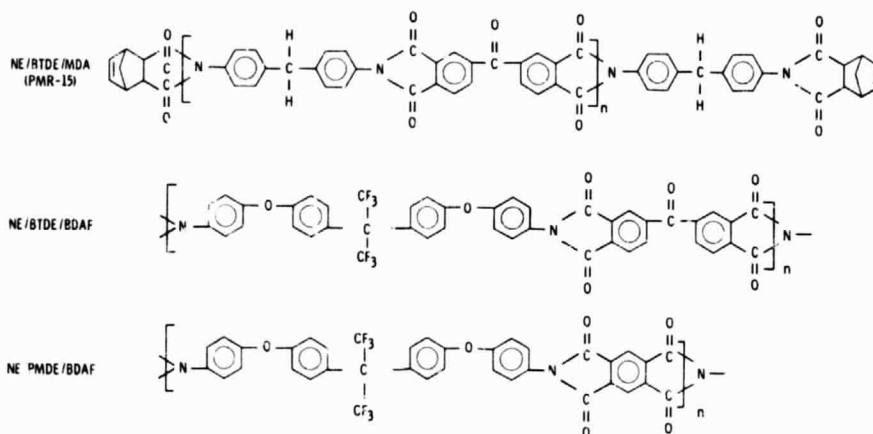


Figure 1. - Repeat unit structures of PMR prepolymers.

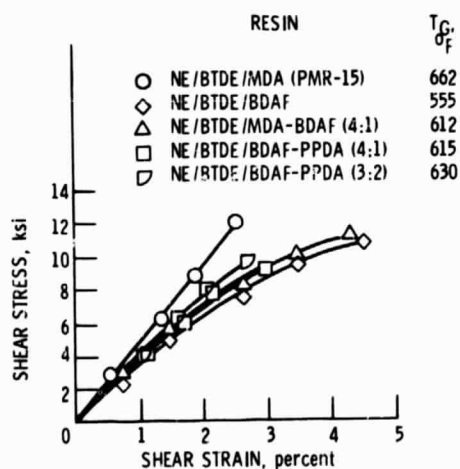


Figure 2. - Ten degree off-axis shear properties of Cation 6000/BDAF-PMR polyimide composites.

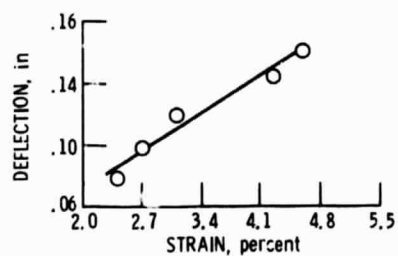


Figure 3. - Ten degree off-axis failure strain vs deflection at maximum drop weight load for Cation 6000/PMR-BDAF composite.

1. Report No. NASA TM-87081		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle Graphite/PMR Polyimide Composites with Improved Toughness				5. Report Date	
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7. Author(s) Raymond D. Vannucci and Kenneth J. Bowles				8. Performing Organization Report No. E-2661	
				10. Work Unit No.	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Memorandum	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes Prepared for the 17th National SAMPE Technical Conference, Kiamesha Lake, New York, October 22-24, 1985.					
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